इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“पुराने को छोड नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

IS 14816 (2000): Leather - Method of Tests for ECO Criteria
[CHD 17: Leather, Tanning Materials and Allied Products]

“ज्ञान से एक नये भारत का निर्माण”
Satyanarayan Gangaram Pitroda
“Invent a New India Using Knowledge”

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
Bhartṛhari—Nītiśatakam
“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

भारतीय मानक

चमड़ा — इको मापदंड की परीक्षण पद्धति

Indian Standard

LEATHER — METHOD OF TESTS FOR ECO CRITERIA

ICS 13.020.50;59.140.30;71.040.40

© BIS 2000

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 2000 **Price Group 7**

Leather Sectional Committee, CHD 17

CONTENTS

Clause	*Subject*	*Page*
LC : 0	General	1
LC : 1	Determination of *p*H and Differential Number of Water Soluble Matter	2
LC : 2	Determination of Hexavalent Chromium in Leather	3
LC : 3	Determination of Formaldehyde (free and released) in Leather	6
LC : 4	Determination of Certain Azo-dyes in Dyed Leather	10

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather Sectional Committee had been approved by the Chemical Division Council.

The environmental impact of leather processing has been a matter of concern among the users as well as in the leather manufacturers. With a growing awareness of environmental aspects, the concept of environment-friendly leather is becoming more and more popular.

Keeping in view the above the Ministry of Environment and Forests, Government of India has decided to include finished leather under the ECO-Mark Scheme. While laying down the ECO criteria for finished leather necessity for test method for the ECO-friendly parameters was felt. The Leather Sectional Committee decided to formulate this Indian Standard on the test method. These test methods are recent and acceptable internationally by and large. There is no ISO Standard on the following subjects. In formulating this standard considerable assistance has been taken from the following documents:

CEN 289008	Leather — Chemical tests — Determination of formaldehyde content in leather
CEN 289055	Leather — Chemical tests — Determination of hexavalent chromium content
CEN 289063	Leather — Chemical tests — Determination of certain Azo-dyes in leather

The method of test for *p*H of leather was already published in LC:18 of IS 582. However the method of test for *p*H and differential number of aqueous extract of leather, given in this standard is identical with ISO 4045:1977 'Leather — Determination of *p*H', published by the International Organization for Standardization (ISO).

The composition of the technical committee responsible for formulating this standard is given at Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

GENERAL

[LC : 0]

1 SCOPE

This standard prescribes the following method of tests for ECO criteria for leather:

Determination of pH and Differential Number of Water Soluble Matter

Determination of Hexavalent Chromium in Leather

Determination of Formaldehyde (free and released) in Leather

Determination of Certain Azo-dyes in Dyed Leather

2 REFERENCES

The Indian Standards listed below contain provisions, which through reference in this text constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
582 : 1970	Methods of chemical testing of leather (*first revision*)
1070 : 1992	Reagent grade water (*third revision*)
5868 : 1983	Methods of sampling for leather (*first revision*)

3 TERMINOLOGY

For the purpose of this standard the following definitions shall apply:

3.1 Certain Azo-dyes — Certain azo-dyes are such dyes as may, by cleavage of their azo group or azo groups, form the amines listed below:

According to the current state of scientific knowledge, evidence of the use of banned azo-dyes in the manufacture or treatment of leathers is said to exist, where the coloured leather yields upon cleavage under the conditions of this procedure (LC:4) one of the following amines and the determined level of any of these amines exceeds 30 mg/kg.

1.	4-Aminodiphenyl	(CAS-No.:92-67-1)
2.	Benzidine	(CAS-No.:92-87-5)
3.	4-Chlor-*o*-toluidine	(CAS-No.:95-69-2)
4.	2-Naphthylamine	(CAS-No.:91-59-8)
5.	*p*-Chloroaniline	(CAS-No.:106-47-8)
6.	2,4-Diaminoanisole	(CAS-No.:615-05-4)
7.	4,4-Diaminadiphenylmethane	(CAS-No.:101-77-9)
8.	3,3-Dichlorobenzidine	(CAS-No.:91-94-1)
9.	3,3-Dimethoxybenzidine	(CAS-No.:119-90-4)
10.	3,3-Dimethylbenzidine	(CAS-No.:119-93-7)
11.	3,3-Dimethyl-4.4'-diaaminodiphenylmethane	(CAS-No.:838-88-0)
12.	*p*-Cresideine	(CAS-No.:120-71-8)
13.	4,4-Methylene-bis-(2-chloroaniline)	(CAS-No.:101-14-4)
14.	4,4-Oxydianline	(CAS-No.:101-80-4)
15.	4,4-Thiodianiline	(CAS-No.:139-65-1)
16.	*o*-Toluidine	(CAS-No.:95-53-4)
17.	2,4-Diaminotoluene	(CAS-No.:95-80-7)
18.	2,4,5-Trimethylaniline	(CAS-No.:137-17-7)

(CAS-No.: Chemical Abstract Substance Registry No.)

According to this method, azo-dyes which yield upon cleavage of their azo groups the amines *o*-aminoazotoluene (CAS-No.:97-56-3) and 2-amino-4-nitrotoluene (CAS-No.:99-55-8), are detected via the amines *o*-toluidine and/or 2,4-diaminotoluene.

3.2 Differential Number (Difference Figure) — The difference between the pH value of a solution and that of its ten-fold dilution. It is a measure of the strength of acids and bases and can never exceed the value 1. The difference figure amounts to 0.7 to 1.0 when a solution contains a free strong acid (or a free strong base). The ionization of weak acids and bases increases with greater dilution, and therefore the difference figure can only act as a criterion for the presence of free strong acid or base in aqueous extracts with pH values below 4 or over 10.

3.3 Hexavalent Chromium Content — The amount of hexavalent chromium in leather determined by this method after extraction with an aqueous salt solution at pH 7.5-8.0. Hexavalent chromium content is reported as hexavalent chromium in mg/kg based on dry matter.

4 QUALITY OF REAGENTS

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

LEATHER — DETERMINATION OF *p*H AND DIFFERENTIAL NUMBER OF WATER SOLUBLE MATTER

[LC : 1]

1 SCOPE

This method prescribes procedure for determining the *p*H and the differential number (difference figure) of an aqueous leather extract.

2 PRINCIPLE

Preparation of an aqueous extract from a test portion of the leather, and measurement of the *p*H of the extract, using a *p*H meter.

3 APPARATUS

3.1 Suitable Shaker — Adjusted to a frequency of 50 ± 10 per minute.

3.2 *p*H Meter with Glass Electrode — With measuring range from 0 to 14 *p*H units, graduated in 0.05 *p*H unit. The electrode system shall be calibrated at intervals against the buffer solution (**4.2**).

NOTE — Aqueous extracts of heavily fat-liquored leather may in time make the electrode membrane dirty. In such cases the membrane should be lightly rubbed with a piece of cotton wool dipped in acetone or the electrode should be suspended in a 1:1 water-acetone mixture. After cleaning, the membrane should again be thoroughly soaked in water.

3.3 Balance — accurate to 0.05 g.

3.4 Wide-mouthed Flask with Leak-proof Stopper — capacity 100 ml.

3.5 Measuring Cylinder — capacity 100 ml, graduated in 1 ml divisions.

3.6 Volumetric Flask — capacity 100 ml.

3.7 Pipette — capacity 10 ml.

NOTE — All glassware shall be made of resistant glass of low alkali content. It is necessary to check the apparatus carefully before use by a blank test using distilled water. The *p*H value and conductivity before and after this test shall be within the limits given in **4.1**. Polyethylene and boroslicate glasses have been found suitable.

4 REAGENTS

4.1 Water — having a *p*H value between 6 and 7 and conductivity not greater than 2×10^{-6} S/cm at 20°C. The water shall be kept in a freshly boiled-out container of resistant glass of low alkali content.

4.2 Buffer Solution — for calibrating the electrode system.

It is preferable to purchase a standard buffer solution for measurement. If purchased in concentrated form, the control buffer must be freshly prepared each time. The length of time for which buffer solutions will keep depends on their composition and the method of use. Control of the accuracy of the buffer solution is therefore indispensable. Used buffer solution shall be discarded.

5 SAMPLING

Samples shall be taken in accordance with IS 5868.

6 PROCEDURE

6.1 Test Portion

Weigh 5 ± 0.1 g of the test sample, prepared in accordance with IS 5868.

6.2 Preparation of the Extract

Place the test portion (**4.1**) in the wide-mouthed flask (**3.4**) and add 100 ± 1 ml of water (**4.1**) at 20 ± 2°C. Shake well by hand for about 30 s, so that the test portion is uniformly wet. Then shake mechanically in the shaker (**3.1**) for 6 h. Allow the extract to settle before decanting. If difficulty is experienced in decanting the extract from the slurry, it may be strained through a clean, dry, non-absorbent mesh (for example, nylon cloth or a coarse sintered glass filter), or centrifuged.

6.3 Determination of *p*H Value

6.3.1 Standardize the *p*H meter with two buffer solutions; one below the expected value and one above the expected value. Both these buffer readings shall be within 0.02 *p*H unit of the correct reading when the meter is standardized.

6.3.2 Bring the temperature of the extract (**6.2**) to 20 ± 1°C. Determine the *p*H value of the extract with the *p*H meter (**6.2**) to the nearest 0.05 *p*H unit, as soon as a steady reading has been reached. The reading shall be taken within 30 to 60 s after rinsing the electrodes in the extract.

6.4 Determination of Differential Number

If the *p*H value is below 4 or over 10, the difference figure shall be determined. For this determination, transfer, using the pipette (**3.7**), 10 ml of the extract into the volumetric flask (**3.6**) and make up to the mark with water. Rinse the electrodes with approximately 20 ml of the diluted solution and then measure the *p*H value as in **6.3**.

7 TEST REPORT

The test report shall include the following particulars:

a) reference to this standard;

b) details of all deviations from the prescribed test conditions;

c) reference to any instability of the *p*H reading of the extract which prevents an unequivocal statement of the *p*H value or difference figure;

d) statement of the mean value of the individual determinations of *p*H value and, if this value is below 4 or over 10, the differential number. The figures should be given to the nearest 0.05 *p*H unit.

DETERMINATION OF HEXAVALENT CHROMIUM IN LEATHER — METHOD OF TEST

[LC : 2]

1 SCOPE

This method prescribes procedure for determination of hexavalent Chromium (C_r^{6+}) in solutions leached from leather under defined conditions.

NOTE — Strongly coloured leather extracts may interfere with the determination. The conditions described in this procedure are suitable for most leathers. In some cases it may be necessary to use a lower or higher cell length or another sample to eluent ratio.

2 PRINCIPLE

Soluble hexavalent chromium is leached from the sample at *p*H 7.5 to 8.0 under an inert gas. The hexavalent chromium in solution oxidizes 1,5-diphenylcarbazide to 1,5-diphenylcarbazone to give a red/violet complex with chromium, which can be quantified photometrically at 540 nm.

The results obtained from the described method are very much dependent on the extraction conditions.

Results obtained by using other extraction procedures (extraction solution, *p*H, extraction time, etc) are not comparable with the results produced by the procedure described in this standard.

3 APPARATUS

3.1 Mechanical Shaker — 50 to 150 revolutions per minute.

3.2 Conical Flask — 250 ml capacity.

3.3 Aeration Tube and Flow Meter

3.4 *p*H-meter — with glass electrode.

3.5 Membrane Filter — 0.45 µm pore size (Teflon or Nylon).

3.6 Volumetric Flask — 50 ml, 100 ml, 1 000 ml.

3.7 Pipettes — nominal volumes 0.5,1.0, 2.0, 5.0, 10, 20, 25 ml.

3.8 Spectrophotometer or Filter photometer — wave length 540 nm.

3.9 Photometric Cell, Quartz — 2 cm length or any other suitable cell length.

3.10 Glass Bottle — with screw cap, 100 ml.

4 REAGENTS

4.1 Extraction Solution — 22.8 g of dipotassiumhydrogenphosphate dissolved in 1 000 ml water, adjusted to *p*H 8.0 ± 0.1 with phosphoric acid (4.3).

4.2 Diphenylcarbazide Solution — 1.0 g of 1,5-diphenylcarbazide is dissolved in 100 ml acetone and made acidic with one drop of glacial acetic acid.

NOTE — The solution should be kept in a brown glass bottle. The shelf life is up to 14 days at 4°C.

4.3 Phosphoric acid Solution — 700 ml of *o*-phosphoric acid (density 1.71 g/ml), made up to 1 000 ml with distilled water.

4.4 Hexavalent Chrome Stock Solution — 2.829 g of potassium dichromate (**4.8**) is dissolved in water in a volumetric flask and made up to 1 000 ml with water. 1 ml of this solution contains 1 mg of chromium (C_r^{6+}).

4.5 Hexavalent Chrome Standard Solution — 1 ml of stock solution (**4.4**) is pipetted in a 1 000 ml volumetric flask and made up to the mark with distilled water. 1 ml of this solution contains 1µg chromium (C_r^{6+}).

4.6 Argon or Nitrogen — oxygen free.

4.7 Distilled Water — *see* IS 1070.

4.8 Potassiumdichromate — dried for 16±2 h at 102±2 °C.

5 PROCEDURE

5.1 Sampling and Preparation of Samples

If possible sample and grind leather in accordance with IS 5868. If sampling in accordance with IS 5868 is not possible (that is leathers from finished products like shoes, garments) details about sampling shall be given together with the test report.

5.2 Preparation of Analytical Solution

Weigh 2 ± 0.01 g of ground leather to the nearest 0.001 g. Pipette 100 ml of degassed solution (**4.1**) into a 250 ml conical flask (**3.2**) and add the leather. Displace oxygen by passing oxygen free argon (or nitrogen) into the flask for 5 min (50 ± 10 ml/min). Remove the aeration tube and close the flask with a stopper of glass, polyethylene or teflon.

The leather powder is extracted by shaking 3 h ± 5 min on a mechanical shaker.

NOTE — Preference should be given to argon as inert gas instead of nitrogen because argon has a higher specific weight than the air while nitrogen has a lower one and leaves the flasks if they are opened.

The settings of the shaking apparatus shall be such that the leather powder is in smooth circular movement without adhering to the wall of the flask. Too fast movement shall be avoided.

After 3 h of extraction check the *p*H of the solution. The *p*H of the solution shall be between 7.5 to 8.0. If it is not within this range the complete procedure shall be started again.

Immediately after the extraction is completed filter the content of the conical flask through a membrane filter into a glass bottle with screw cap.

NOTE — Some very acidic wet blues may lower the *p*H of the eluent below 7.5. It may also happen in single case that the *p*H is higher than 8.0. In such cases the sample weight shall be reduced during the repeat test so that the *p*H of the extract is within 7.5-8.0.

5.3 Determination of Hexavalent Chromium in the Solution

5.3.1 Pipette 10 ml of the solution obtained through 5.2 into a 50 ml volumetric flask. Dilute the solution to 3/4th of the flask's volume with distilled water. Add 1 ml of diphenylcarbazide solution **(4.2)** and afterwards 1 ml of phosphoric acid **(4.3)**. Mix the content of the flask and make up to 50 ml with distilled water. Allow it to stand for 15 ± 5 min.

5.3.2 Measure the extinction of the solution at 540 nm in a 2 cm cell against the blank solution **(5.4)**. Record the extinction obtained as E_1.

5.3.3 Pipette another 10 ml aliquot of the solution into a 50 ml volumetric flask and treat it as described in **5.3.1** but without the addition of the diphenylcarbazide solution. Measure and record the extinction of this solution as E_2.

NOTE — The described procedure may not be suitable for all kinds of leathers. Strongly coloured extracts can interfere with the determination. The procedure is normally applicable up to an extinction (E_2) of the extract of 0.500 (determined in a 2 cm cell at 540 nm). In some of such cases a dilution of the extract and the use of photometric cells with higher or lower cell length may solve these problems. Inter-lab tests have shown that in most cases a 2 cm cell normally produces the best results.

5.4 Blank Solution

Fill three quarters of a 50 ml volumetric flask with distilled water, add 1 ml of diphenylcarbazide solution (5.2) and 1 ml of phosphoric acid (4.3) make up to the mark and mix well. Store this solution in a dark and cool place.

5.5 Calibration

Prepare calibrating solutions from the standard solution **(4.5)** obtained through the chromium concentration in these solutions should cover the expected range of measurements.

The calibrating solutions are prepared in 50 ml volumetric flasks.

Plot a suitable calibration curve by using 0.5, 1.0,3.0, 5.0, 8.0, 10.0, 15.0, 20.0 ml of the standard solution **(4.5)**. This would cover a range of 1.25 to 50.0 mg/kg based on leather. Pipette the given volumes of standard solution **(4.5)** into 50 ml volumetric flasks, 1 ml diphenylcarbazide solution **(4.2)** and add 1 ml of phosphoric acid **(4.3)** to each flask. Make up to volume with distilled water, mix well and allow to stand for 15 ± 5 min.

Measure the extinction of the solutions in a 2 cm cell at 540 nm against the blank obtained in **5.4**.

The hexavalent chromium concentration in mg/50 ml is plotted against the extinctions measured. The hexavalent chromium concentration is plotted on the *x*-axis, the extinction on the *y*-axis.

NOTE — In inter-laboratory tests the 2 cm cell proved to be most suitable. In some cases it may however suitable to use higher or lower cell length.

5.6 Determination of Recovery Rate

The determination of the recovery rate is important to provide information about possible matrix effects, which can influence the results.

Pipette 10 ml aliquot of the solution obtained in **(5.2)** into a 50 ml volumetric flask. Add suitable volume of standard solution **(4.5)** to double the content of the hexavalent chromium concentration of the extract with a tolerance 25 percent.

After the addition of the standard solution, add 1 ml of diphenylcarbazide solution **(4.2)** and 1 ml of phosphoric acid **(4.3)** is added and mix the solution.

After 15 ± 5 min measure the extinction of the solution at 540 nm against the blank solution **(5.4)**. Record the obtained extinction as E_3.

The extinction of the solution shall be within the range of the calibration curve, otherwise the procedure is repeated by using a smaller aliquot (that is 5 ml).

NOTES

1 If added hexavalent chromium can not be detected this may be an indication that the leather contains reducing agents. In some cases, after intensive considerations, this may lead to the conclusion that this leather has no hexavalent chromium content (below detection limit).

2 The recovery rate is an indicator whether the procedure works or whether matrix effects are influencing the results. Normally the recovery rate is more than 70 percent.

6 CALCULATION

6.1 Calculation of Hexavalent Chromium Content

$$W_{Cr^{6+}} = \frac{(E_1 - E_2) \times V_1 \times V_2 \times 10^3}{50 \times A \times m \times F}$$

$W_{Cr^{6+}}$ = Soluble hexavalent chromium in leather in mg/kg,

E_1 = Extinction of sample solution with diphenylcarbazide,

E_2 = Extinction of sample solution without diphenylcarbazide,

F = Gradient of calibration curve (y/x),

A = Aliquot taken from eluate (ml),

m = Original mass of leather taken (g),

V_1 = Extraction volume (in this standard 100 ml), and

V_2 = Volume to which the aliquot was made up (in this standard 50 ml)

Result based on dry matter:

$$W_{Cr}^{6+}\,\text{dry} = W_{Cr}^{6+} \times D$$

D is the factor for conversion to dry matter

$$D = \frac{100}{100 - W}$$

W = Volatile matter determined using the method prescribed in LC:1 of IS 582.

DETERMINATION OF FORMALDEHYDE CONTENT IN LEATHER — METHOD OF TEST

[LC : 3]

This method prescribes two procedures of test for the determination of free and released formaldehyde in leathers. One method is known as HPLC method and the other as photometric method. The first method is selective and not sensitive to coloured extracts.

1 HPLC METHOD

1.1 Scope

Formaldehyde contents are understood to be the quantity of formaldehyde contained in the extract of the leather under the following conditions referred to the weighed-in quantity of leather:

a) formaldehyde is separated and quantified as derivative from other aldehydes and ketones by liquid chromatography;

b) free formaldehyde; and

c) formaldehyde, which is hydrolzed during extraction to yield free formaldehyde.

1.2 Principle

The leather sample is eluted with water at 40°C. The eluate is mixed with dinitrophenylhydrazine, whereby aldehydes and ketones react toward the respective hydrazons. The solution is separated by means of HPLC-reverse phase, detected at 350 nm and quantified.

1.3 Apparatus

1.3.1 *Strainer with Glass Fibre Filter* — 90 g/m^2 (or glass filter strainer G3, diameter 7-10 cm).

1.3.2 *Witt's Pot* — (*see* Fig. 1).

1.3.3 *Water Bath* — with possibility to stir or wave, thermostatically controlled to 40 ± 0.5°C.

1.3.4 *Thermometer* — with 0.1°C graduation for the range 20-50°C.

1.3.5 *HPLC with UV Detection* — 350 µm.

1.3.6 *Membrane Filter* — nylon, 0.45 µm.

1.4 Reagents

Unless otherwise stated only analytically pure chemicals shall be used. The water shall be distilled or of reagent grade (*see* IS 1070). Solution shall be understood to be an aqueous solution.

1.4.1 *Sodiumdodecyl Sulfonate* — 1g of in 1 000 ml water.

1.4.2 *Sulphuric Acid* — concentration, 1.5 ml/l (3N).

1.4.3 *Sodium Hydroxide* — concentration 2 mol/l (1N).

1.4.4 *Sodium thiosulphate Solution* — concentration 0.1 mol/l (0.1N).

1.4.5 *Iodine Solution* — concentration 0.05 mol/l (0.1N), 12.68 g iodine.

1.4.6 *Starch Solution* — concentration 1 g/100 ml.

1.4.7 *Dinitrophenylhydrazine Solution (DNPH)* — concentration 0.3 percent in concentrated hydrochloric acid (recrystallized from 25 percent acetonitril/water)

1.4.8 *Formaldehyde Solution* — concentration approximately 37 percent.

1.4.9 *Acetonitril*

1.5 Preparation and Standardization of Formaldehyde Stock Solution

Pipette 5 ml of formaldehyde solution into a 1 000 ml volumetric flask which contains approximately 100 ml water and subsequently fill with water up to the mark. From this solution, pipette 10 ml each into a 250 ml Erlenmeyer flask and mix with a 50 ml iodine solution (**1.4.5**) as well as with sodium hydroxide (**1.4.3**) until it turns yellow. Allow it to settle for 15 minutes at 22±4 °C and then add 50 ml of sulphuric acid (**1.4.2**) while wheeling about. Upon addition of 2 ml of starch solution, titrate the excess iodine with sodium thiosulphate (**1.4.4**) until colour changes. Make 3 individual determinations and carry out a blank test in the same manner.

Calculation of the content of the formaldehyde stock-solution:

$$C_{FA} = \frac{(V_0 - V_1) \times C_1 \times M_{FA}}{2}$$

where

C_{FA} = Concentration of the formaldehyde stock solution in mg/10 ml;

V_0 = Volume of the used thiosulphate solution of the blank solution in ml;

V_1 = Volume of the used thiosulphate solution of the measuring solution in ml;

M_{FA} = Molecular weight of formaldehyde (30.08) g/mol; and

C_1 = Concentration of thiosulphate solution in mol/l.

1.6 Determination of Formaldehyde in Leather

1.6.1 *Extraction and DNPH Reaction*

Weigh accurately (to 1 mg) 2 g of leather into a 100 ml Erlenmeyer flask with 29/32 socket. Add 50 ml solution (**1.4.1**) (pre-warmed to 40°C) and close the flask with a glass stopper). Stir the content of the flask smoothly at 40 $\pm$ 0.5°C in a water bath for 1 hour $\pm$ 2 minutes. Subsequently filter the still warm extract solution through a glass fibre filter into a 100 ml flask by means of a Witts Pot (*see* Fig. 1). Cool down the filtrate with closed flask to room temperature (22 $\pm$ 4°C). Pipette in a 10 ml volumetric flask 4 ml acetonitril and also an aliquot of 5 ml of the eluate and 0.5 ml DNPH solution (**1.4.7**). Fill it with water up to the mark and mix. After at least 60 min, and maximum upto 180 minutes the sample, will be chromatographed the sample upon filtration through a membrane filter (Nylon 0.45 m). If the concentration is out of the calibration range take smaller aliquotes.

NOTE

1 Leather /solution ratio shall not be changed.

2 Standard addition procedure is recommended.

1.6.2 *HPLC Conditions (Recommendation)*

Flow rate	1.0 ml/minute
Mobile phase	acetonitril : water : : 60 : 40
Separation column	Merck 100, CH 18,2 (highly coated, 12 percent C) + pre-column (1cm) (RP 18)
UV detection	350 nm
Injection volume	20 µl

1.7 Calibration

Pipette 0.5 ml of the formaldehyde solution obtained in **1.6.1** with exactly known content into a 500 ml volumetric flask, pre-filled with approximately 100 ml water, mix, fill with water upto the mark, and again mix well (Content approximately 2 µg/ml).

This solution is the standard solution.

Fill in each of 10 ml volumetric flasks, 4 ml acetonitril and prepare a concentration series of 0,5; 1; 2; 3; 4; 5 ml of standard solution. Mix with each content of the volumetric flasks, immediately upon addition of the formaldehyde solution and add 0.5 ml DNPH solution (**1.4.7**) to each, fill up to the mark with water and mix thoroughly. After at least 60 minutes and maximum before 180 min, chromatograph the sample after filtration through a membrane filter. The calibration is effected through protraction of the surface unit of the formaldehyde derivative versus the concentration in µg/10 ml.

1.8 Calculation of Formaldehyde Content in Leather

$$C_F = \frac{C_S \times F}{E_W}$$

where

C_F = Concentration of formadehyde in the sample in mg/kg rounded to 0.01 mg/kg,

C_S = Concentration of formaldehyde obtained from the calibration in µg/10 ml,

F = Dilution factor, and

E_W = Quantity in-weighed in g.

1.9 Spiking

Fill 4 ml acetonitril into a 10 ml volumetric flask and add an aliquot of 2.5 ml of the filtrate as obtained from **1.6**. Add an accurately determined volume of the formaldehyde standard solution, which will lead to an almost equal concentration as found in the sample.

Treat this solution further analogous to the description under **1.6**, and C_{S2} determined analogous to **1.8**. Double determination with description of individual values, shown on notification sheet.

$$RR = \frac{(2 \times C_{S2} - C_S) \times 100}{2 \times C_{FAI}}$$

where

RR = Recovery rate in percent, rounded to 0.1 percent.

C_{S2} = Concentration of formaldehyde obtained from the calibration in µg/10 ml,

C_S = Concentration of formaldehyde in the non-spiked sample in µg/ml,

C_{FAI} = Spiked quantity of formadehyde in µg/10 ml, and

2 PHOTOMETRIC METHOD

2.1 Scope

Formaldehyde contents are understood to be the quantity of formaldehyde contained in the extract of the leather under the following conditions referred to the weighed-in quantity of leather. The process is not absolutely selective. Free formaldehyde and formaldehyde, which is hydrolized during extraction to yield free formaldehyde, are detected in this method.

2.2 Principle

The leather sample is diluted with water at 40°C. The eluate is treated with acetylacetone, whereby formaldehyde reacts to give a yellow compound (3,5-diacetyl-1,4-dihydrolutidine). The absorption of this compound is measured at 412 nm. The amount of formaldehyde corresponding to the determined absorbance is obtained from a calibration curve prepared under identical conditions.

2.3 Reagents

2.3.1 *Sodium Dodecyl Sulfonate* — 1 g of in 1 000 ml water (to be freshly prepared daily).

2.3.2 150g of ammonium acetate with 3 ml of glacial acetic acid and 2 ml of acetylacetone (Pentane-2,4-dion; CAS-No. : 123-54-6) in 1 000 ml water

2.3.3 150g of ammonium acetate with 3 ml glacial acetic acid in 1 000 ml water

2.3.4 5g dimedone in 1 000 ml water (dimedone= Methone=5,5-Dimethyl-1.3-cyclohexadion; CAS-No. : 126-81-8)

NOTE — It is reported that dimedone is not dissolved in pure water. In such cases dimedone can be dissolved in a small amount of ethanol and then filled up with water.

2.4 Preparation and Standardization of Formaldehyde Stock Solution

2.4.1 *Reagents* — The quality of reagents shall be in accordance with **1.4**.

2.4.2 *Procedure* — The standard solution shall be prepared in accordance with **1.5**.

2.5 Procedure for Determination of Formaldehyde in Leather

2.5.1 *Extraction* — Weigh accurately (to 1 mg) 2 g of leather into a 100 ml Erlenmeyer flask with 29/32 socket. Add 50 ml solution (**1.4.1**) (pre-warmed to 40°C) and close the flask with a glas stopper). Stir the content of the flask smoothly at 40 ± 0.5°C in a water bath for 1 hour ± 2 minutes. Subsequently filter the still warm extract solution through a glass fibre filter into a 100 ml flask by means of a Witts pot (Fig. 1) through glass fibre. Cool down the filtrate with closed flask to room temperature (22 ± 4°C). The leather/solution ratio shall not be modified.

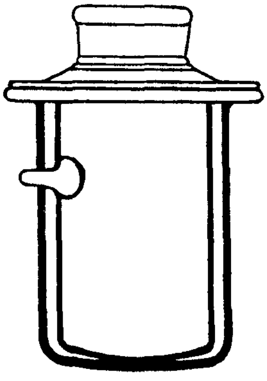

FIG. 1 WITT'S POT

2.5.2 *Derivatization with Acetylacetone* — Pipette 5 ml of the filtrate obtained in **2.5.1** into a 25 ml Erlenmeyer flask and add 5 ml of the reagent solution (**2.5.2**). Fit the Erlenmeyer flask with a glass stopper. Stir the solution for 30 min at a temperature of 40±1°C. After cooling under exclusion of light measure the extinction at 412 nm versus 5 ml solution (**2.3.1**) and 5 ml solution (**2.3.2**). Record the extinction obtained as E_{pr}.

For the purpose of determining the extinction conditioned on the self-colouring of the filtrate obtained in **2.5.1**, pipette 5 ml of the filtrate (**2.5.1**) into a 25 ml Schlifferlenmeyer flask and add 5 ml solution (**2.3.3**). Thereafter, apply the same method as was done with the sample. The extinction obtained will be registered as E_e.

NOTE — Take a smaller aliquot for leathers with a high content of formaldehyde (>75 mg/kg). Smaller aliquots than 5 ml are filled up with the difference to 5 ml with water.

Example : Formaldehyde content : 500 mg/kg

Procedure : Pipette 0.5 ml of the filtrate (2.5.1) in a 25 ml Erlenmeyer flask. Add 4.5 ml of water. Then follow the procedure as described above.

2.5.3 *Checking Reagents for Absence of Formaldehyde* — Measure 5 ml of solution (**2.3.1**) and 5 ml reagent (**2.3.2**) in relation to 5 ml of solution (**2.3.1**) and 5 ml of water. The measured extinction shall not be larger than 0.025 (measured in a 2 cm cell at 412 nm).

2.5.4 *Testing Other Compounds Which Cause Colouring with Acetylacetone* — Mix 5 ml of filtrate obtained in **2.5.1** with 1 ml dimedone solution (**2.3.4**), warm upto 40°C for 10 min and after adding 5 ml reagent solution (**2.3.2**) warm upto 40°C for 30 min. Upon cooling down to room temperature, take a measurement is at 412 nm versus a blank solution, which instead of the filtrate contains 5 ml of reagent solution (**2.3.1**). At this measurement, no extinction (< 0.05) shall be existing when formaldehyde was existing at the first measurement.

2.6 Calibration — Pipette 3 ml of the formaldehyde stock solution obtained in **2.4.2** with exactly known amount into a 1 000 ml volumetric flask which has been pre-filled with 100 ml water, mix and fill with water up to the mark and again thoroughly mix (standard approx 6 µg/ml). This solution is the standard solution.

From this solution pipette 3, 5, 10, 15, 25 ml each, into a 50 ml volumetric flask and fill up with water. These solutions cover the range of 0.4 µg/ml to 3.0 µg/ml. (Corresponds to a concentration range of 9-74 mg/kg leather-under the given conditions). With regard to leather at higher concentrations a smaller aliquot of filtrate is used.

From these solutions, pipette 5 ml each and mix in a 25 ml Erlenmeyer flask (with 29/32 socket) with 5 ml solution (**2.3.2**). Shake this mixture intensively and warm upto 40 ± 1°C for 30 minutes.

Upon cooling down to room temperature (protect against light) take a measurement at 412 nm versus a solution consisting of 5 ml (**2.3.2**) and 5 ml of water.

Prior to measurement, the zero point of the photometer calibrate with the blank sample [5 ml (**2.3.2**) and 5 ml water], which was treated under the same conditions as the measuring solution versus water.

The concentrations in µg/ml shall be protracted versus the measured extinction. (x-axis: concentration in µg/ml, y-axis: extinction).

2.7 Calculation of the Content of Formaldehyde in Leather Sample

$$C_p = \frac{(E_p - E_e) \times V_o \times V_f \times 1\,000}{F_x \times W_x \times V_a}$$

where

C_p = Concentration of formaldehyde in the sample in mg/kg, rounded off to 0.1 mg/kg;

E_p = Extinction of filtrate after reaction with acetylacetone;

E_e = Extinction of filtrate (self-colouring);

V_o = Volume of elution in ml (standard conditions : 50 ml);

V_a = Aliquot taken from filtrate in ml (standard conditions : 5 ml) (*see* Note);

V_f = Volume of solution obtained in (**2.5**) after derivatization in ml (standard conditions : 10 ml);

F = gradient of calibration curve ($\Delta y/\Delta x$) in ml/µg; and

W = weight of leather in g.

NOTE — For interlab test smaller aliquot are required.

2.8 Spiking, Recovery Rate

Pipette 2.5 ml (*see* Note) of the filtrate obtained in **2.5.1** in a 10 ml volumetric flask. To the content of the volumetric flask add an exactly determined volume of the formaldehyde solution, which will lead to the approximately same concentration as was found in the sample. Fill the volumetric flask with water up to the mark.

Transfer the content of the volumetric flask to a 25 ml Erlenmeyer flask, add 5 ml of reagent solution (**2.3.2**) and stir for 30 min at 40 ± 1°C.

After having cooled down, take a measurement of the extinction at 412 nm versus a 5 ml solution (**2.3.1**) and 5 ml solution (**2.3.2**). Record the extinction of the increased sample as E_A.

$$RR = \frac{(2E_A \times - E_p) \times 100}{2 \times E_{ZU}}$$

where

E_A = Extinction of the spiked sample

E_p = Extinction of the non-spiked sample

E_{ZU} = Expected extinction of that where the quantity of formaldehyde was added (from calibration)

RR = Recovery rate in percent, rounded off to 0.1 percent.

Example: Found in the leather sample: 30 mg/kg — Increase with 5 ml of the formaldehyde standard solution.

NOTE — If the formaldehyde content is found below 20 mg/kg, take 5 ml aliquot instead of 2.5 ml.

DETERMINATION OF CERTAIN AZO-DYES IN DYED LEATHER — METHOD OF TEST

[LC : 4]

1 SCOPE

This method prescribes procedure for determination certain azo-dyes, which may release certain aromatic amines.

2 PRINCIPLE

After degreasing, the leather sample is treated with sodium dithionite in an aqueous buffer solution (*p*H 6) at 70°C in a closed vessel. The amines released in the process of reductive cleavage are transferred to a *t*-butyl methyl ether phase by means of liquid-liquid extraction using Kieselghur columns. The *t*-butyl methyl ether extract is then concentrated under mild conditions in a rotary vacuum evaporator and the residue is dissolved in a suitable solvent, depending on the method used to determine the amines.

Determination of the amines is performed by means of high pressure liquid chromatography using a diode array detector (HPLC/DAD), thin layer chromatography with a flame ionization detector and/or a mass specific detector (GC/FID and/or MSD), or by capillary electrophoresis with a diode array detector (CE/DAD).

The amines are identified by means of at least two different chromatographic separation methods in order to avoid any possible misinterpretations caused by interfering substances (such as position isomers of the amines to be identified) and hence any incorrect statements. Amine quantification is performed by HPLC/DAD.

3 APPARATUS

3.1 Suitable Reaction Vessel — made of temperature-resistant glass with gas tight closure.

3.2 Hot Cabinet with Sand Bath — (sea sand of particle size 0.1-0.3 mm) or water bath with thermostat.

3.3 Thermometer — 0.1°C accuracy at 70°C.

3.4 Volumetric Flasks — of different volumes.

3.5 Polypropylene or Glass Column — with 25 to 30 mm inner diameter and 140 to 150 mm length, gas filter at the outlet, filled with porous granulated Kieselghur.

3.6 Polypropylene or Polyethylene Syringe — 2 ml.

3.7 Vacuum Rotary Evaporator

3.8 Pipettes — 10 ml, 5 ml, 2 ml, 1 ml.

3.9 Ultrasonic Bath with Thermostat

3.10 Round-bottom Flask with Standard-Ground Joint — 100 ml.

3.11 Analytical Instruments

- Automatic applicator for HPLC or DC
- Densitometer
- Capillary electrophoresis with DAD
- Capillary GC, split/splitless injector, preferably with MS/MSD
- HPLC with gradient controller, preferably with DAD, or HPLC-MS.

4 REAGENTS

4.1 Methanol

4.2 *t*-Butyl Methyl Ether

4.3 Sodium Dithionite — minimum 87 percent purity.

4.4 Aqueous Sodium Dithionite Solution — Concentrated 2 000 mg/ml, freshly prepared.

4.5 *n*-Hexane

4.6 Amines — 1 to 18 of **3.1** of the procedure (highest available purity standard).

4.7 Stock-Solution of the Amines — 6.6 : 400 µg/ml in ethyl acetate for DC.

4.8 Stock-Solution of the Amines — 6.6 : 200 µg/ml in methanol for GC, HPLC,CE.

4.9 Citrate Buffer Solution — 0.06 M, *p*H 6 (preheated to 70°C).

4.10 Standard Solution for Amine — process control: 1 30 µg/ml amine/ml solvent.

4.11 20 percent Methanolic Sodium Hydroxide — 20 g sodium hydroxide dissolved in 100 ml methanol.

5 PREPARATION OF SAMPLES

Depending on the sample, grind the leather sample, if this is not possible, the sample is cut into square pieces with a side length of 3 ± 0.2 mm and subsequently mixed. For the analytical procedure, weigh a representative sample of 1.0 g of this mixture into the reaction vessel (**3.1**).

Remove traces of adhesives, where necessary, mechanically.

6 PROCEDURE

6.1 Degreasing

Treat 1g of the leather sample in a closed 50 ml vessel (**3.1**) with 20 ml *n*-hexane in an ultrasonic bath at 40°C for 20 minutes. Decant the *n*-hexane layer from the leather sample. Treat the sample directly afterwards again in the same way as before with 20 ml *n*-hexane. Evaporate the residue *n*-hexane overnight in the open vessel.

6.2 Reductive Cleavage

Add a quantity of 17 ml buffer solution (**4.4**) preheated to 70 ± 5°C to the sample. Seal the reaction vessel tightly, and after shaking, keep it in a ventilated oven in a sand bath or on a water bath for 25 ± 5 min at 70 ± 2°C. The reaction temperature of 70°C shall be reached inside the reaction vessel. Check this with an additional vessel with thermometer inside.

Then add 1.5 ml aqueous sodium dithionite solution with a syringe and keep the vessel for 10 min at 70°C. Afterwards add another 1.5 ml sodium dithionite solution and the vessel is heated for another 10 min and cooled to room temperature with water.

6.3 Liquid-Liquid-Extraction

Using a glass pestle, squeeze the reaction solution out of the fibres, decant on the Kieselghur column and allow it to be absorbed by the column for 15 min. Add 5 ml of *t*-butyl-methyl ether and 1 ml of 20 percent methanolic NaOH to the leather fiber residue in the vessel. Close the vessel, shake vigorously and transfer to the Extrelut column.

Then elute the amines with 1 × 15 ml and 1 × 20 ml *t*-butyl methyl ether by washing the reaction vessel and the fiber residue. Evaporate the remainder of the ether to dryness using a slight flow of inert gas.

Transfer the residue immediately to a 2 ml volumetric flask and make up the volume with solvent. This solution is ready for the instrumental analysis.

6.4 Check of the Analytical System

To check the procedure of analysis, add 1.0 ml of the calibration solution (**4.10**) to a reaction vessel (**3.1**) containing 16 ml of the preheated buffer. Then carry out the procedure set out in **8.2**. The recovery rate shall be at least 70 percent for the amines: 1-5, 7-15, and 18, at least 50 percent for amines 16,17 and at least 20 percent for amine 6.

7 CALIBRATION

The standard solutions with 15 µg/ml are used for calibration.

8 CHROMATOGRAPHIC ANALYSIS

As various type of equipment may be used, general statements cannot be made. The following parameters have been successfully tested and used in these analysis.

8.1 Chromatographic Analysis for Quantitative and Qualitative Detection

High Performance Liquid Chromatography (HPLC)

Eluent 1 : Methanol

Eluent 2 : 0.575g of Ammonium di-hyrogen phosphate and 0.7g of disodium hydrogenphosphate in 1 000 ml water, *p*H 6.9

Stationary phase : LiChrospher 60 RP-select B (5 µm)

Column : 250 × 4.6 mm

Temperature : 40 °C

Flow rate : 0.8 - 1.0 ml/min

Gradient : Start 15 percent eluent 1, linear increase to 80 percent eluent 1 within 45 min

Injection volume : 10 µl

Detection : DAD at 240 nm, 280 nm and 305 nm

8.2 Chromatographic Analysis for Qualitative Detection

8.2.1 *Capillary Gas Chromatography (GC)*

Capillary column : Medium polarity, that is SE 54 or DB5, length: 50 m, inner diameter 0.32 mm, film thickness: 0.5 µm

Injection system : Split/splitless

Injector temperature : 250°C

Temperature programme : 70°C for minutes upto 280°C with 10°C/min. 280°C for 5 min

Detector : MSD, scan 45-300 amu

Carrier gas : Helium

Injection : 1 µl, splitless 2 minutes

8.2.2 *Capillary Electrophoresis (HPCE)* — Mix 250 µl of the sample solution with 50 µl CCI (concentration 0.01 mol/l) and pass through a membrane filter (0.2 µm). Analyze this solution by means of capillary zone electrophoresis.

Capillary 1 : 56 cm, uncoated, inside diameter: 50 µm, with extended light path

Capillary 2 : 56 cm, coated with polyvinyl alcohol (PVA), inside

Diameter : 50 µm, with extended light path

Buffer solution : Phosphate buffer solution (concentration 50 mol/l), *p*H 2.5

Column temperature	:	25°C
Voltage	:	30 kV
Injection time	:	4 sec
Flushing time	:	5 sec
Detection	:	DAD 214 nm, 240 nm, 280 nm, 305 nm

8.2.3 *Thin Layer Chromatography (TLC)*

Plates (HPTLC)	:	Silica gel with fluorescence indicator F 254, 20 × 10 cm
Applied volume	:	5 μl applied a line with automatic applicator
Mobil solvent 1	:	Chloroform: ethyl acetate : acetic acid = 60:30:10 parts per volume
Mobil solvent 2	:	Chloroform: methanol = 95:5 parts per volume;
Reagent 1	:	0.1 percent $NaNO_2$ in KOH (concentrated 1 mol/l)
Reagent 2	:	0.2 percent or Naphthol, in KOH (concentrated 1 mol/l)

9 EVALUATION

Calculate amine concentration based on the peak areas of the individual amine components. The content of the amine is calculated as mass portions W in mg individual component/kg leather according to the following equation:

$$W_{\text{amine component}} = \frac{A_p \times \beta \times V}{A_k \times E}$$

where

A = peak area of the amine in area units

β = concentration of the amine in the calibration solution in μg/ml,

V = volume the sample is made up to according to **6.3** (final sample volume) in ml,

E = weight portion accounted for by the leather sample in the final sample volume in g, and

W = Mass portion.

Indices :

P = Sample, and

K = Calibration solution of amine.

10 ANALYSIS REPORT

Any analysis report shall refer to this analytical procedure and state the following particulars:

- kind, origin and designation of the analyzed article or part thereof;
- any deviations from the analytical procedure, particularly any additional steps performed; and
- declaration of the performed separation procedure and methods used for detection and determination (minimum 2).

10.1 In the Case of Levels per Amine Component is not more than 30 mg/kg

According to the analysis as carried out, azo-dyes which release the listed aromatic amines were not detected.

10.2 In the Case of Levels per Amine Component is more than 30 mg/kg

The analysis result suggests that the leather submitted has been manufactured or treated by using azo-dyes which release one or more of the listed amines.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Leather Sectional Committee, CHD 17

Chairman	*Representing*
Dr T. Ramasami	Central Leather Research Institute, Chennai
Members	
Shri Anil Agarwal Shri M. K. Awasthi (*Alternate*)	Ministry of Defence (R & D)
Shri Shafeeque Ahmed Shri V. P. Naimur Rahman (*Alternate*)	Indian Finished Leather Manufacturers and Exporters Association, Chennai
Shri S. K. Basu	Department of Education, Govt of West Bengal, Calcutta
Shri S. K. Basu	Development Commissioner, Small Scale Industries, New Delhi
Shri O. P. Behari Dr N. R. Mukhopadhyay (*Alternate*)	Directorate of (Marketing Inspection Ministry of Agriculture), Faridabad
Shri S. K. Bhadra	Indian Leather Technologists' Association, Calcutta
Director (Leather)	Khadi and Village Industries Commission, Mumbai
Shri P. K. Ghosh Shri N. K. Mazumdar (*Alternate*)	Bata India Ltd, Calcutta
Shri G. Gupta Shri G. C. Kanaujiya (*Alternate*)	Ministry of Defence (DGI), DGQA, Kanpur
Shri O. K. Kaul Shri R. Subhramaniam (*Alternate*)	Tata International Ltd, Dewas
Shri P. D. Mayee Shri S. C. Lahiry (*Alternate*)	Planning Commission, New Delhi
Shri P. V. Parvitikar	Footwear Design & Development Institute, Noida
Shri S. Raja	Leatherware, Bangalore
Representative	Council for Leather Exports, Chennai
Representative	Leather Research and Testing Laboratory, Driectorate of Industries, Kanpur
Representative	National Institute of Fashion Technology, New Delhi
Shri A.K. Satwah Shri P. Jayakumaran (*Alternate*)	Directorate General of Supply and Disposal, New Delhi
Shri Ajay Shankar Shri Sanjeev Gupta (*Alternate*)	Ministry of Defence (DGOF), Kanpur
Shri G. M. Sharif	All India Small Scale Tanners and Exporters' Association, Chennai
Shri M. Srinivas Shri N. M. Zachariah (*Alternate*)	All India Skins and Hides Tanners and Merchants' Association, Chennai
Shri Lajinder Singh, Director (Chem)	Director General, BIS (*Ex-officio Member*)

Member-Secretary
Shri P. Mukhopadhyay
Additional Director (Chem), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act,* 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. **CHD 17 (898).**

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	323 76 17 323 38 41
Eastern	: 1/14 C. I.T. Scheme VII M, V. I. P. Road, Kankurgachi CALCUTTA 700 054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160 022	60 38 43 60 20 25
Southern	: C. I. T. Campus, IV Cross Road, CHENNAI 600 113	235 02 16, 235 04 42 235 15 19, 235 23 15
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400 093	832 92 95, 832 78 58 832 78 91, 832 78 92

Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM.

Printed at : Prabhat Offset Press, New Delhi-2